

## Research paper

# High-speed and high-quality TSV filling with the direct ultrasonic agitation for copper electrodeposition



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## ABSTRACT

In this paper, the influential factors of the silent and ultrasonic electroplating using 3-mercaptopropanesulfonate (MPS), polyethylene glycol (PEG), and Polyethylenimine alkyl salt (PN) on the TSV filling are investigated. The effects of different accelerator concentration, current density and ultrasonic agitation on TSV filling are studied. The accelerative effect of MPS on TSV filling by the copper electrodeposition is studied to find out the optimal condition of the accelerator concentration. Using the plating bath with same additive conditions, the effects of different current densities on the TSV filling under the silent and ultrasonic conditions are investigated. The results show that the quality of TSV filling under ultrasonic electroplating has improved by 23% compared with the silent condition at the same current density.

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## 1. Introduction

Three-dimensional (3D) packaging technology is crucial for modern micro-assembly technology, and plays a key role in the field of microelectronics technology [1–5]. The 3D packaging technology can be divided into three main types: embedded 3D packaging, active substrate-based 3D packaging, and stacked 3D packaging [6–10]. The through silicon via (TSV) interconnection is an advantageous technique for 3D packaging, as it can provide high stack density, high efficiency, and fast communication due to the fact that it provides vertical interconnection between components [11–13].

In order to meet the reliability requirement for TSV, the void-free TSV-filling should be achieved in its manufacture process, which has been a focus for many researchers [14–18]. Many investigations have been made on the effect of different additives added into the electroplating bath [19–23]. In addition, the external conditions of the impact should also not be ignored, such as the applied pulse reverse current, applied ultrasound, the forced convection, environment temperature, etc., which have greatly effects on the mass transfer in the microstructure. Jin et al. [24] used the high-frequency pulse current to realize the TSV filling. Chen et al. [25] studied the effects of ultrasonic agitation, mechanical agitation, pulse current and DC on the TSV filling. Effects of forced convection on the TSV-filling process have been studied by Zhang et al. [26]. High-frequency ultrasonic application to plating to get a lower diffusion layer was proposed by Kaufmann et al. [27], and the fluid flow in vias under high-frequency ultrasound is investigated

by Costello et al. [28]. Nevertheless, the effect of ultrasound on the TSV-filling has not investigated deeply enough and the mechanisms of the ultrasound applied in the TSV-filling process are unrevealed.

In this paper, effect of ultrasonic plating on the electroplating of TSV is studied, which mainly focused on the three parameters of accelerator concentration, current density and with or without ultrasonic. The comparison of the TSV filling without and with ultrasound at the same condition is presented. Ultrasonic improvements are confirmed, and the dynamic profiles of the cross-sectional TSV under ultrasonic field are obtained. In addition, the velocity field induced by the acoustic streaming in the plating bath is analyzed.

## 2. Experiments

Copper electroplating apparatus mainly consists of four parts: a plating bath (e.g. Coase Lynn electroplating instrument company), a cathode and an anode, and an ultrasonic source device (e.g. FS-300N with frequency 20 kHz from Shanghai sonxi ultrasonic instrument co., LTD), as shown in Fig. 1. The plating chamber's length, width, and height are 24 cm, 62 cm, and 12.5 cm, respectively. In the ultrasonic experiment process, ultrasonic source is placed between the cathode and anode. Phosphor copper anode is fixed in the plating bath on the left side of the plating chamber. The small chip with microvias as the cathode is fixed approximately 4.5 cm from the anode in Haring cell.

The copper electroplating solution is composed of copper sulfate (195 g/L), sulfuric acid (0.32 g/L), 3-mercaptopropanesulfonate (MPS) as an accelerator (varying concentration), polyethylene glycol (PEG) as a suppressor (1.5 g/L), and Polyethylenimine alkyl salt (PN) as a leveler (0.02 g/L). In the experiment, the circular vias with an aspect

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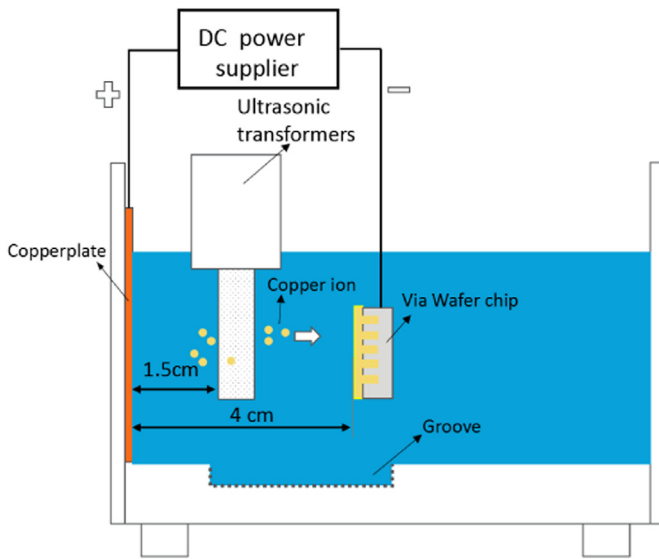


Fig. 1. An electroplating system for TSV filling under ultrasonic agitation.

ratio of 3 (60  $\mu\text{m}$  in depth and 20  $\mu\text{m}$  in width) in the silicon chip (size of 0.8 cm  $\times$  1.2 cm) are used. In order to deposit copper into the microvias successfully, the internal surface of the blind vias in the purchased silicon chip are already covered with a thin copper layer as a seed layer (about 1  $\mu\text{m}$ ). The chip is pretreated as follows. The piece of TSV chip is first fixed vertically in a beaker containing deionized water. The beaker is then put into the vacuum pump with the pressure kept at  $-15$  psi for five minutes. To eliminate the bubbles in the vias, the ultrasonic cleaning machine is utilized to vibrate and rotate the beaker rapidly for 5–10 s. The aforementioned pretreatment is repeated for three to four times, until no bubbles are observed on the surface of the TSV. The TSV chip is placed in electroplating solution for 10 min, so that the solution can be fully transported into blind-vias. The precise power supplier provides a constant current. The filled TSV is sealed vertically in epoxy resin which is placed in the ambient air for 5 h for drying. Then the sealed sample is grinded and polished, and the impurities on the surface of the sample are cleaned. Finally, scanning electron microscopy images of the filled TSV are taken for further analysis.

Table 1

The parameters of accelerator concentration, the current density and ultrasound condition in experiments.

Experiment number	MPS concentration (mL)	Current density (ASD)	Ultrasonic
A1	0	0.2	Off
A2	0.5	0.2	Off
A3	1	0.2	Off
A4	1.5	0.2	Off
A5	2	0.2	Off
B1	1	0.1	Off
B2	1	0.2	Off
B3	1	0.3	Off
B4	1	0.4	Off
B5	1	0.5	Off
B6	1	0.6	Off
B7	1	0.7	Off
B8	1	0.8	Off
C1	1	0.1	On
C2	1	0.2	On
C3	1	0.3	On
C4	1	0.4	On
C5	1	0.5	On
C6	1	0.6	On
C7	1	0.7	On
C8	1	0.8	On

Three groups (group A, group B, group C) of TSV filling experiments are conducted and the main parameters are given in Tables 1. Group A shows the parameters of TSV filling experiments to obtain the optimal accelerator concentration. Group B gives the parameters for the study of the different current densities without ultrasound. The parameters for the study of the different current densities with ultrasound are shown in Group C.

### 3. Results and discussion

#### 3.1. The deposition performance without ultrasound

In this paper, a certain additive combination is studied. In this combination, accelerators and suppressor is considered no interaction (such as replacement reaction of the suppressor by the accelerator) on the surface. Because adsorption coefficient of the suppressor is large and the diffusion coefficient of the suppressor is small, the suppressor is mainly distributed in the TSV orifice for the copper deposition inhibition, while the accelerator is just the opposite. Considering the same assumption, our previous work [29] presented the model of TSV filling which is validated by the experiment. This paper is investigated on the combination of a sufficient concentration of suppressor. It is observed that under the sufficient concentration of suppressor (1.5 g/L), the concentration of the accelerator (MPS) and the current density are the critical conditions to decide the TSV-filling quality. The effects of different accelerator concentrations on copper electrodeposition are studied to obtain the optimal condition. Five experiments are implemented under the current density of 0.2 ASD using 0 mL, 0.5 mL, 1 mL, 1.5 mL and 2 mL of the accelerator solution (concentration of 10 g/L) into the 0.9 L solution, respectively. Fig. 2 shows the images of the cross section of the blind-vias using different concentration of the accelerator. When the dose of the accelerator is 0 mL, it is clear that seams form in the middle of the via; when it increases to 0.5 mL, the seams disappear and the via could be fully filled by the electrodeposition of copper; when it further increases to 2 mL, seams appear again. The possible explanation is that the higher concentration of the accelerator leads to a stronger deposition of copper in the via, and the suppressor at the top inhibits the deposition. According to our previous study, for the sufficient concentration of suppressor (1.5 g/L), the increase of inhibitors did not reduce the seams of TSV. If the accelerator concentration is extremely high, the speed of deposition will increase tremendously, and the inhibition from the suppressor is less effective on the top, thus leading to the appearance of seams. Blind-vias could be fully filled using the accelerator dose between 0.5 mL to 1.5 mL. Therefore, the accelerator of around 1 ml dose in this situation is the preferred concentration.

The effects of different current densities on the TSV filling in the absence of ultrasound at 1 mL dose of the accelerator are studied. The filling results using current density ranging from 0.1 ASD to 0.8 ASD after 5 h of plating is illustrated in Fig. 2. It can be seen that, when the current density is 0.1 ASD and 0.2 ASD (Fig. 4a and b), the vias can be fully filled. But with the increase of current density, a small void appears near the bottom of the via. When the current density increases from 0.3 ASD (Fig. 4c) to 0.8 ASD (Fig. 4h), the volume of the filling void increases quickly, and the included angle at the top of the void becomes larger. When the current density is 0.8 ASD, the worst filling quality is obtained. The experimental results indicate that the change of current density affects the deposition rate of copper electrodeposition and the TSV-filling quality. The patterns can be summarized that, the larger the current density is, the faster the opening part grows and the larger void in the via is formed.

#### 3.2. Effect of ultrasound on the TSV filling

In this section, the impact of ultrasound on electroplating by applying different current densities under the same plating bath is analyzed and the results are illustrated in Fig. 4. In this paper, the ultrasound of

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